



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁷ : G01N 35/00	A1	(11) International Publication Number: WO 00/45180 (43) International Publication Date: 3 August 2000 (03.08.00)
(21) International Application Number: PCT/US99/12686 (22) International Filing Date: 7 June 1999 (07.06.99) (30) Priority Data: 60/118,225 1 February 1999 (01.02.99) US 09/245,949 5 February 1999 (05.02.99) US (71) Applicant: 3M INNOVATIVE PROPERTIES COMPANY [US/US]; 3M Center, P.O. Box 33427, Saint Paul, MN 55133-3427 (US). (72) Inventors: KO, John, H.; P.O. Box 33427, Saint Paul, MN 55133-3427 (US). PETERSON, James, R.; P.O. Box 33427, Saint Paul, MN 55133-3427 (US). (74) Agents: ROGERS, James, A. et al.; 3M Innovative Properties Company, Office of Intellectual Property Counsel, P.O. Box 33427, Saint Paul, MN 55133-3427 (US).		(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: POLY(ALPHA-OLEFIN) ADHESIVE COVER TAPES FOR ANALYTICAL RECEPTACLES (57) Abstract <p>An analytical receptacle that includes a surface having at least one reservoir therein and a cover tape adhered to the surface. The cover tape comprises a backing and an adhesive coated on at least one major surface of the backing, wherein the adhesive comprises a poly(alpha-olefin) polymer.</p>		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece			TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	NZ	New Zealand		
CM	Cameroon			PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakhstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	SG	Singapore		

POLY(ALPHA-OLEFIN) ADHESIVE COVER TAPES FOR ANALYTICAL RECEPTACLES

The invention relates to cover tapes for analytical receptacles, such as
5 microliter plates, microfluidic devices, discrete or continuous multi-reservoir carriers, or
other analytical receptacles. The invention particularly relates to cover tapes for analytical
receptacles that are designed for holding a variety of materials, particularly liquids, in bio-
analytical applications, for example.

Microtiter plates are well known for use in handling liquid materials in bio-
10 analytical assays for multiple, rapid, low-volume analysis. A typical screening technique
combines an assay plate, having multiple depressions or wells, with liquid handling
hardware to provide a rapid, automated system of analysis. In a current, standard
analytical system, each assay plate accommodates 96 wells, each well being addressable
by suitably programmed hardware. The capacity of each of the 96 wells is approximately
15 0.5 milliliter (ml). Smaller capacity wells lead to assay plates that accommodate a larger
number of samples. For example assay plates containing 1536 wells, each with a capacity
of approximately 1 microliter (μ l) are known. These plates, with increased sampling
capability, have demonstrated usefulness in a variety of assays, including enzyme assays,
receptor-ligand assays, and even cell based assays. The increased number of sample wells,
20 per assay plate, demands increased precision of the hardware associated with analysis
using these assay plates.

Liquid handling for bio-analytical applications, using assay plates of either
the 96-well, 384-well, or the 1536-well variety, may be viewed as a batch process with rate
limitation due to the loading and positioning of the assay tray. Possible improvement in
25 the rate of sample analysis results from the use of a continuous strip of material having
sample wells molded along its length. U.S. Pat. No. 4,883,642 (Bisconte) suggests such a
strip or tape. The patent teaches a continuous ribbon, which may be either smooth or
suitably molded to incorporate a plurality of micro-wells. Fixed biological sample
analysis uses smooth versions of the continuous ribbons while micro-well ribbons find use
30 for analyzing living biological samples. Two tracks, positioned along opposite edges of
the ribbon, provide addressable means for moving and positioning the ribbon in a selected,

precise location with adjustment accurate to 10 micrometers (μm). The tracks may be coded using magnetic, optical, or computer methods, for example, which allow manipulation and positioning of the ribbon. A dosage syringe type of device, positioned using a step by step motor, distributes biological samples in the microwells.

5 This continuous multi-reservoir carrier is useful in the automatable analysis of biological samples, such as histological sections. Protection of the samples, whether applied to a smooth ribbon or contained in micro-wells may use a self-adhesive film. The self-adhesive film covers the smooth film surface or seals the openings to the individual micro-wells. It may be permeable or impermeable to air.

10 What is needed are cover tapes such as this that provide an effective seal on analytical receptacles, particularly cover tapes that are substantially resistant to liquids, particularly organic solvents such as dimethyl sulfoxide that are often used in bio-analytical applications.

15 The present invention provides cover tapes for analytical receptacles, such as microliter plates, microfluidic devices, and continuous multi-reservoir carriers, or other analytical receptacles or biosensors, for example. Typically, such analytical receptacles are used in bio-analytical applications and are designed for containing solids and fluids, including liquids, gases, powders, and gels, which may include biological samples or organic solvents, for example.

20 The cover tapes for such analytical receptacles provide a sealing membrane so that each reservoir, such as a well or channel, for example, is part of a sealed enclosure to retain the contents and reduce evaporation and contamination of the contents of the receptacle. The cover tapes of the present invention preferably have good optical clarity and are preferably substantially resistant to solvents commonly used in bio-analytical
25 applications, such as dimethyl sulfoxide (DMSO), water, acetonitrile, methanol, ethanol, or mixtures thereof, for example. As used herein, a substantially resistant adhesive is one that does not substantially swell or dissolve in the solvent used in the particular application and does maintain sufficient adhesion to the analytical receptacle.

30 In one embodiment, the present invention provides an analytical receptacle comprising a surface having at least one reservoir therein and a cover tape adhered to the surface; wherein the cover tape comprises a backing and an adhesive coated on at least one

major surface of the backing and in contact with the receptacle surface; wherein the adhesive comprises a poly(alpha-olefin) polymer.

The analytical receptacle can be in the form of a substantially continuous tape or it can be in discrete shapes and sizes. For example, the analytical receptacle can be in the form of a microliter plate, a microfluidic device comprising a substrate and one or more channels therein, or a substantially continuous polymeric strip (i.e., tape) comprising a plurality of reservoirs at predetermined intervals (preferably, uniformly spaced) along its length. Thus, the reservoir(s) can be in a wide variety of shapes and sizes. Preferably, they form wells or channels.

The adhesive can be pattern coated on the backing of the cover tape or it can form a continuous layer on at least one major surface of the backing. It is preferably a pressure sensitive adhesive. The adhesive may or may not be crosslinked, although preferably it is crosslinked. This crosslinking may occur using a photoactive crosslinking agent, although this is not required with E-beam radiation.

The present invention provides cover tapes for analytical receptacles, such as assay plate arrays (e.g., microliter plates) and discrete or continuous (e.g., strip or tape) structures containing a plurality of wells, channels, or other reservoirs. The analytical receptacle, without further modification, provides an open system of one or more reservoirs (e.g., wells or channels) to which fluids may be added directly. Open systems require careful control of evaporation and cross-contamination, which limits their practical applications. Thus, cover tapes are desirable as they result in closed systems that do not necessarily require specialized sample transport and containment.

A cover tape is applied along the length and width of an analytical receptacle to seal the reservoirs) of the receptacle. Preferably, this results in producing individually sealed enclosures. Materials may be injected into or extracted from the closed reservoirs, through the cover tape, using suitable hypodermic-type needles, for example, if so desired. The analytical receptacles can include one or more reservoirs. They can be substantially continuous or discrete (i.e., noncontinuous) structures. For example, an analytical receptacle can be in the form of a microliter plate that is conventionally used in bio-analytical methods. Alternatively, it can be a microfluidic device or continuous multi-

precise location with adjustment accurate to 10 micrometers (μm). The tracks may be coded using magnetic, optical, or computer methods, for example, which allow manipulation and positioning of the ribbon. A dosage syringe type of device, positioned using a step by step motor, distributes biological samples in the microwells.

5 This continuous multi-reservoir carrier is useful in the automatable analysis of biological samples, such as histological sections. Protection of the samples, whether applied to a smooth ribbon or contained in micro-wells may use a self-adhesive film. The self-adhesive film covers the smooth film surface or seals the openings to the individual micro-wells. It may be permeable or impermeable to air.

10 What is needed are cover tapes such as this that provide an effective seal on analytical receptacles, particularly cover tapes that are substantially resistant to liquids, particularly organic solvents such as dimethyl sulfoxide that are often used in bio-analytical applications.

15 The present invention provides cover tapes for analytical receptacles, such as microliter plates, microfluidic devices, and continuous multi-reservoir carriers, or other analytical receptacles or biosensors, for example. Typically, such analytical receptacles are used in bio-analytical applications and are designed for containing solids and fluids, including liquids, gases, powders, and gels, which may include biological samples or organic solvents, for example.

20 The cover tapes for such analytical receptacles provide a sealing membrane so that each reservoir, such as a well or channel, for example, is part of a sealed enclosure to retain the contents and reduce evaporation and contamination of the contents of the receptacle. The cover tapes of the present invention preferably have good optical clarity and are preferably substantially resistant to solvents commonly used in bio-analytical
25 applications, such as dimethyl sulfoxide (DMSO), water, acetonitrile, methanol, ethanol, or mixtures thereof, for example. As used herein, a substantially resistant adhesive is one that does not substantially swell or dissolve in the solvent used in the particular application and does maintain sufficient adhesion to the analytical receptacle.

30 In one embodiment, the present invention provides an analytical receptacle comprising a surface having at least one reservoir therein and a cover tape adhered to the surface; wherein the cover tape comprises a backing and an adhesive coated on at least one

major surface of the backing and in contact with the receptacle surface; wherein the adhesive comprises a poly(alpha-olefin) polymer.

The analytical receptacle can be in the form of a substantially continuous tape or it can be in discrete shapes and sizes. For example, the analytical receptacle can be in the form of a microliter plate, a microfluidic device comprising a substrate and one or more channels therein, or a substantially continuous polymeric strip (i.e., tape) comprising a plurality of reservoirs at predetermined intervals (preferably, uniformly spaced) along its length. Thus, the reservoir(s) can be in a wide variety of shapes and sizes. Preferably, they form wells or channels.

The adhesive can be pattern coated on the backing of the cover tape or it can form a continuous layer on at least one major surface of the backing. It is preferably a pressure sensitive adhesive. The adhesive may or may not be crosslinked, although preferably it is crosslinked. This crosslinking may occur using a photoactive crosslinking agent, although this is not required with E-beam radiation.

The present invention provides cover tapes for analytical receptacles, such as assay plate arrays (e.g., microliter plates) and discrete or continuous (e.g., strip or tape) structures containing a plurality of wells, channels, or other reservoirs. The analytical receptacle, without further modification, provides an open system of one or more reservoirs (e.g., wells or channels) to which fluids may be added directly. Open systems require careful control of evaporation and cross-contamination, which limits their practical applications. Thus, cover tapes are desirable as they result in closed systems that do not necessarily require specialized sample transport and containment.

A cover tape is applied along the length and width of an analytical receptacle to seal the reservoirs) of the receptacle. Preferably, this results in producing individually sealed enclosures. Materials may be injected into or extracted from the closed reservoirs, through the cover tape, using suitable hypodermic-type needles, for example, if so desired. The analytical receptacles can include one or more reservoirs. They can be substantially continuous or discrete (i.e., noncontinuous) structures. For example, an analytical receptacle can be in the form of a microliter plate that is conventionally used in bio-analytical methods. Alternatively, it can be a microfluidic device or continuous multi-

reservoir carrier, for example, which can be cut into discrete (noncontinuous) pieces, if desired.

A cover tape of the present invention, which acts as a sealing membrane, includes a poly(alpha-olefin) adhesive, preferably, a pressure sensitive adhesive, disposed on a backing. Preferably, the backing is made of a transparent material. A cover tape of the present invention adheres well to low energy and cold surfaces with desired wetstick and repositionability, but does not allow cross-contamination of sample materials in the individual reservoirs. Preferably, the cover tape maintains adhesion during high and low temperature storage (e.g., about -80°C to about 150°C) while providing an effective seal against sample evaporation. Suitable cover tapes of the present invention allow for puncture by needles, such as stainless steel needles, or plastic sampling pipette tips, for example, although cover tapes that resist puncture can also be used.

Because use of the cover tape can expose the adhesive to fluid contents of a reservoir, the choice of adhesive is of particular importance. Conventional adhesive tapes frequently use acrylate adhesive compositions, which dissolve or otherwise react with solvents, such as dimethyl sulfoxide (DMSO) and acetonitrile, used commonly in bio-analytical research. Thus, preferred adhesives are substantially resistant to a wide variety of solvents, such as DMSO, water, acetonitrile, methanol, ethanol, or similar polar solvents, as well as mixtures thereof. That is, preferred adhesives do not substantially swell or dissolve in the solvent used in the particular application while they maintain sufficient adhesion to the analytical receptacle.

Furthermore, preferred adhesives are pressure-sensitive adhesives with sufficiently low tack that they leave little or no residue on the analytical receptacles or the needles or pipette tips after withdrawal from the puncture hole in the cover tape. This low tack also typically prevents adhesion of the cover tape to commonly used rubber gloves made from latex or nitrile rubber. As used herein, a pressure-sensitive adhesive has a four-fold balance of adhesion, cohesion, stretchiness, and elasticity with an open tack time (i.e., period of time during which the adhesive is tacky at room temperature) on the order of days and often months or years, and a glass transition temperature of less than about 20°C.

Preferred adhesives also are substantially biocompatible (i.e., substantially physiologically inert). As used herein, a "biocompatible" material is one that does not

generally cause significant adverse reactions (e.g., toxic or antigenic responses) when in contact with biological fluids and/or tissues, such as tissue death, tumor formation, allergic reaction, inflammatory reaction, or blood clotting, for example.

The adhesive used in the cover tapes of the present invention includes a poly(alpha-olefin). Poly(alpha-olefins) have excellent adhesion, thermaloxidative stability, and chemical resistance. For example, poly(alpha-olefin) polymers have good solvent resistance to, for example, water, methanol, ethanol, acetonitrile, and DMSO. Furthermore, they are substantially physiologically inert. These properties make them excellent adhesives for use in cover tapes for analytical receptacles that are used in bio-analytical applications. Examples of suitable adhesives containing a poly(alpha-olefin) polymer are disclosed, for example, in U.S. Pat. Nos. 5,112,882 (Babu et al.), 5,859,088 (Peterson et al.), 5,407,970 (Peterson et al.), 3,542,717 (Lipman), and 3,635,755 (Balinth).

A preferred adhesive includes a radiation curable poly(alpha-olefin)-containing adhesive composition that is pressure-sensitive at room temperatures, which is disclosed in U.S. Pat. No. 5,112,882 (Babu et al.). The composition includes at least one polymer prepared from monomers including: about 85 mole percent to about 100 mole percent, preferably, about 85 mole percent to about 99 mole percent, of one or more C6 to C10 alpha-olefin monomers, and about 15 mole percent to 0 mole percent, preferably, about 15 mole percent to about 1.0 mole percent, of one or more polyene monomers, with the proviso that the mole percentages of all monomers sum to 100. Polymers formed from polyene monomers contain residual ethylenically unsaturated groups in the polymer backbone and/or, preferably, in pendant side chains that can be used in radiation initiated crosslinking reactions to improve the cohesive strength of the adhesive.

Another preferred adhesive includes a saturated poly(alpha-olefin) containing adhesive composition, which is disclosed in U.S. Pat. No. 5,859,088 (Peterson et al.). The composition includes at least one polymer prepared from monomers including: (1) about 70 to about 99 mole percent of one or more C6 to C12 alpha-olefin monomers; and (2) about 1.0 to about 30 mole percent of one or more C2 to C5 alpha-olefin monomers.

Preferably, adhesive compositions containing poly(alpha-olefin) polymers useful for the present invention have a glass transition temperature (Tg) of about -70°C

to about 0°C (more preferably, about -60°C to about -20°C). Preferably, the poly(alpha-olefin) polymers have an inherent viscosity of about 0.4 dl/g to about 9.0 dl/g (more preferably, about 0.5 dl/g to about 6.0 dl/g, and most preferably, about 1.5 dl/g to about 4.0 dl/g), and a number average molecular weight of about 5,000 to about 50,000,000 (more preferably, about 50,000 to about 5,000,000 and most preferably, about 500,000 to about 3,000,000).

The adhesive compositions can include an effective amount of a photoactive crosslinking agent to crosslink the composition upon irradiation from a source of actinic radiation (e.g., UV), although a photoactive crosslinking agent is not a necessary requirement for crosslinking the adhesive composition with E-beam radiation. Typically, for the adhesives of the present invention, crosslinking is optional, although preferred, particularly for reduced extractables and for reduced residue on either needles or pipette tips that puncture the cover tape. Examples of suitable crosslinking agents are disclosed in U.S. Pat. No. 5,112,882 (Babu et al.) and include, for example, aldehydes, ketones, quinones, thioxanthenes, and certain chromophore-substituted vinyl halomethyl-sym-triazines such as 2,4-bis-(trichloromethyl)-6-(3',4'-dimethoxyphenyl)-sym-triazine.

The adhesive composition can also include a tackifying resin, particularly a substantially hydrogenated tackifying resin as disclosed in U.S. Pat. No. 5,112,882 (Babu et al.). Preferably, for reduced residue obtained on needles and pipette tips that may puncture the cover tape the adhesive composition includes a tackifying resin. The adhesive composition preferably includes up to about 100 parts, and more preferably, up to about 50 parts, per 100 parts polymer of a tackifying resin.

The adhesive composition may include other additives to adjust for desired properties. For example, a silicone lubricant, such as Masterbatch MB50-002 (available from Dow Corning, Midland, Michigan), can be added to the adhesive composition mixture in order to reduce the amount of adhesive residue transferred to needles and pipette tips that puncture the cover tape. The adhesive composition includes preferably up to about 5% silicone lubricant and more preferably up to about 1% silicone lubricant by weight, based on the total weight of the adhesive composition. Another example of an additive is a thermoplastic, such as high density polypropylene and polyethylene, which can be added to the adhesive composition to increase the storage modulus, glass transition

temperature, and adhesive strength of the adhesive composition. The adhesive composition includes preferably up to about 25% by weight, and more preferably up to about 10% by weight, based on the total weight of the adhesive composition.

The adhesive composition can be applied to appropriate backings by a wide range of processes, including, solution coating, solution spraying, hot-melt extrusion, emulsion coating, etc., to make adhesive cover tapes.

In the context of the adhesive used in the present invention: "polymer" means a homopolymer, a copolymer, a terpolymer, or a tetrapolymer; polymers derived from more than one monomer may be either random or block polymers; "polyene monomer" means a C4 - C24 hydrocarbon containing two or more conjugated or non-conjugated ethylenically unsaturated groups which may also contain 1 to 5 unitary heteroatoms selected from the group consisting of oxygen and silicon; "C6 to C12 alpha-olefin monomer" means a linear or branched hydrocarbon having 6 to 12 carbon atoms, one terminal ethylenically-unsaturated group, and no other functional group; and "photoactive crosslinking agent" means a compound which, under the influence of radiation, connects polymer chains and becomes incorporated therein, which increases the molecular weight of the adhesive and thus its cohesive strength without unduly affecting its compliance or other PSA properties.

Conventional cover tapes for microliter plates include an adhesive layer and a backing such as aluminum (Al) foil or polyethylene terephthalate (PET). Al foil backings are less desirable because they are not transparent. PET tape backings have high mechanical strength and resist puncture by all but the hardest, sharpest needles. A plastic pipette tip, for example, requires high force to break through a very thin (approximately 1 mil) PET backing.

Suitable backings for use in the cover tapes of the present invention allow for puncture by needles or plastic sampling pipette tips, for example, although the puncture sites of such backings do not reclose. Alternatively, suitable backings that resist puncture can also be used. Preferably, the backing will puncture without splitting. The backings can be transparent, translucent, or opaque. Preferably, the backing is transparent. Transparency facilitates chemical analysis conducted by any one of several methods of photometric analysis including, for example, ultraviolet, visible, and fluorometric analysis.

The backing can include a wide range of substrate materials, examples being polymer films such as polyethylene, polyethylene terephthalate (PET), biaxially oriented polypropylene (BOPP), and metallocene-polymerized poly(alpha-olefin) copolymers. These backing materials are generally resistant to solvents commonly used in bio-analytical applications, as discussed above with respect to the adhesives. They can resist puncture or not, although if they are punctured, the puncture site does not reclose.

The analytical receptacles to which the cover tapes can be applied include a wide variety of articles. Generally, the analytical receptacles include at least on surface having one or more reservoirs therein. For example, a suitable analytical receptacle to which a cover tape of the present invention can be applied includes a microliter plate, which is typically a plastic plate containing a number of small flat-bottomed wells arranged in rows. Another example is a tape that includes a substrate coated with a gel having a plurality of separate adjacent tracks thereon, as disclosed in U.S. Pat. No. 3,551,295 (Dyer).

Other analytical receptacles include microfluidic devices that include a substrate and one or more channels therein. Such a structure, which includes a body structure and at least one microscale channel disposed therein, is disclosed in U.S. Pat. No. 5,842,787 (Kopf-Sill et al.). Yet another such structure, which has a groove recessed in a flat substrate and defines a microfluidic channel system, is disclosed in U.S. Pat. No. 5,443,890 (Ohman). Yet another such structure, which includes a substrate with microstructures fabricated therein, is disclosed in U.S. Pat. No. 5,804,022 (Kaltenbach et al.).

Another type of analytical receptacle includes a substantially continuous polymeric strip formed to have wall portions defining a series of identical reservoirs at predetermined, preferably, uniformly spaced, intervals along its length, which reservoirs can have a variety of shapes. For example, the reservoirs may comprise rectangular or generally "I" or "T" shapes in the plane of the strip, and may have flat or rounded bottoms as desired. Such receptacles are disclosed, for example, in U.S. Pat. No. 4,883,642 (Bisconte). Other such receptacles are disclosed in U.S. Pat. No. 5,729,963 (Bird), which are of the type designed for carrying electrical components, for example, but can be modified for use as analytical receptacles.

These analytical receptacles can be formed from a variety of materials. Examples include, but are not limited to, polyethylene, polystyrene, polypropylene, polycarbonate, as well as carbon-black or TiO₂ filled materials. These materials can be transparent, translucent, or opaque.

5 Objects and advantages of this invention are further illustrated by the following examples, but the particular materials and amounts thereof recited in these examples, as well as other conditions and details, should not be construed to unduly limit this invention.

10 Test Methods

Peel Force: Using an Imass peel tester at 12 inches/minute, according to ASTM D3330-D3330M-96, the 180° peel force was measured after applying the cover tape to planar surfaces of polypropylene test plates.

15 Solvent Resistance: The adhesive tapes were adhered to an aluminum well filled with DMSO for 24 hours. The tapes were subsequently inspected visually for evidence of swelling or dissolution.

Residual Adhesive on Probe Puncture Test: The tapes were adhered to a microplate (available from Corning Inc., Corning, NY). The cover tape was punctured five
20 consecutive times at different locations with either a 22 gauge needle or a 10 µl plastic tip (available from Eppendorf Corp., Germany). The barrel of the needles and tips were visually inspected (10x magnification) for evidence of adhesive residue.

Extractables Test: 4cm x 5cm samples of each tape were extracted with 5 mL of each solvent for 24 hours. The extracts were examined by gas chromatography mass
25 spectroscopy GC/MS using the following conditions:

Instrument: Finnigan "Magnum" GC/ion trap MS

Column: Phenomenex ZB-5 30 mx 0.25mm 0.1 µ

Carrier: Helium at ca 1 mL/minute

Injector: Atlas Optic 2

30 Mode: Cold split, multi-capillary liner

Injector: Vol: 4mL

Inj. Temp. Prog: 30°C/s to 350°C

Pressure Prog: 10 psi initial to 20 psi at 16 minutes

Oven Prog: 50°C @ 20°C/min to 320°C (2.5 minutes)

Transfer Line: 300°C

5 Detector: E1 scan 31 -- 550 Da, no mass defect 1s/scan [3 μ scans] 220°C

Examples 1-20 and Comparative 1 and 2

10 Poly(alpha-olefin) - 100 parts using either poly 1-octene ("Oct") of inherent viscosity 4.1 dl/g or poly (1-octene/ethylene) copolymer ("Oct-E") of inherent viscosity 2.8 dl/g.

Hydrocarbon tackifier REGALREZ 1126 ("Reg") available from Hercules Inc., Wilmington, DE, was added at varying levels as shown in Table 1. Masterbatch (MB50-002) silicone lubricant from Dow Corning, Midland, Michigan was added at the level noted in Table 1.

15 Polypropylene Escorez 3505 available from Exxon, Houston, TX was added to the level noted in Table 1.

Examples 1-20, shown in Table 1, were melt mixed using a Prep-mixer available from G.W. Brabender Co. Chicago, IL. Mixing conditions were defined as a
20 bowl temperature of 180°C, screw speed 60 rpm, and duration of 10 minutes. The adhesive compositions were coated at 0.005 cm thick on a 0.005 cm thick embossed high density polyethylene (HDPE) film backing (available from Bloomer Plastics, Bloomer, WI) using a Haake-modified extruder operating at die temperature 177°C. As noted in
25 Table 1, the coated film of examples 1-18 was then passed through an electron beam chamber to cure the poly(alpha-olefin) adhesive at three different E-beam intensities of 3, 6, and 9 Mrads at 175 meV. For examples 19 and 20, the coated films were not E-beam cured.

The tapes were applied over a polypropylene microplate (available from Dow Corning, Corning, NY), with DMSO in the micro-wells, for 24 hours under ambient
30 conditions. There was no noticeable change of adhesion of the cover tapes of the invention to the polypropylene plate as compared to a comparative nitrile rubber tape

(Minnesota Mining and Manufacturing Co., St. Paul, MN) and a comparative PET tape
(Marsh Laboratories, Pittsburgh, PA).

Table 1. Evaluation of Poly(alpha-olefin) Tapes with E-Beam Curing

Example	Formulation	E-beam Dosage	Peel Adhesion (oz/in)	Adhesion in DMSO (24 hours)	Residue 22 G Needle	Residue 10 μ L tip
1	Oct-E (IV 2.8)	3 Mrad	>50	----		
2	Oct-E (2.8), 25% Reg	3 Mrad	>50	----		
3	Oct-E (2.8), 50% Reg	3 Mrad.	4.0	----		
4	Oct (IV 4.1)	3 Mrad	21.8	----		
5	Oct (4.1), 25% Reg	3 Mrad	12.5	----		
6	Oct (4.1), 50% Reg	3 Mrad	14.0	----		
7	Oct-E (IV 2.8)	6 Mrad	>50	----		
8	Oct-E (2.8), 25% Reg	6 Mrad	>50	----		
9	Oct-E (2.8), 50% Reg	6 Mrad	15.7	Ok*	no	No
10	Oct (IV 4.1)	6 Mrad	38.3	Ok	no	No
11	Oct (4.1), 50% Reg	6 Mrad	22.6	Ok	no	No
12	Oct (4.1), 50% Reg	6 Mrad	13.4	Ok		
13	Oct-E (IV 2.8)	9 Mrad	>50	----	no	Slight
14	Oct-E (2.8), 25% Reg	9 Mrad	>50	----	no	No
15	Oct-E (2.8), 50% Reg	9 Mrad	7.9	Ok		
16	Oct (IV 4.1)	9 Mrad	29.3	Ok		
17	Oct (4.1), 25% Reg	9 Mrad	12.4	Ok	no	No
18	Oct (4.1), 50% Reg	9 Mrad	12.4	----	no	No
19	Oct (2.9) with 1% MB50-002	0 Mrad	18.9	Ok	no	No
20	Oct (2.9) with 25% Reg, 10% Polypropylene	0 Mrad	3.7	Ok	no	No
Comp. 1	Nitrile rubber	----	----	Swollen,		

Example	Formulation	E-beam Dosage	Peel Adhesion (oz/in)	Adhesion in DMSO (24 hours)	Residue 22 G Needle	Residue 10 μ L tip
	tape			lost adhesion		
Comp. 2	Marsh PET tape	----	----	Swollen, lost adhesion		

*no noticeable change of adhesion.

Examples 21-24

Poly 1-octene ("Oct") and a UV initiator (2,4-bis-(trichloromethyl)-6-(3',4'-dimethoxyphenyl)-sym-triazine) as disclosed in US Pat. No. 4,330,590 (Vesley) were mixed in the amounts shown in Table 2.

Compositions of Examples 21-24 were coated on the corona treated side of a 1.2 mil biaxially oriented polypropylene film backing, using a Haake-modified extruder to provide coating weights of 1.5-2.0 mils. The extruder operated at 177°C. After coating, the tape was UV-irradiated at 316 mJ/cm² to provide a crosslinked layer having the desired adhesive properties.

Table 2. Poly(alpha-olefin) Compositions with UV Curing

Examples	% Triazine	Peel Adhesion (oz/in)	Adhesion in DMSO 24 Hours
21	0	47.0	ok
22	0.1	17.0	ok
23	0.15	18.0	ok
24	0.20	----	ok

Examples 25-26 and Comparative 3-6

Examples 25 and 26 were compounded and coated as in Examples 19 and 20. Comparative 3 was Corning 3095 cover tape available from Dow Corning Corp., Corning, NY. Comparative 4 was PN 62367 cover tape available from Zymark Corp., Hopkinton, MA. Comparative 5 was Costar 6569 cover tape available from Corning Costar Corp., Acton, MA. Comparative 6 was Ultra-Plate cover tape available from Sagian Corporation, Indianapolis, IN.

Table 3. Extractable study of Poly(alpha-olefin) Adhesive Compositions

Examples	Tape or PSA Description	Extractant	Total Extracts, $\mu\text{g}/\text{cm}^2$
25	Oct (2.9) with 25% Reg, 10% Polypropylene	DMSO	5-20
25	Oct (2.9) with 25% Reg, 10% Polypropylene	80/20, AN*/H ₂ O	2-5
25	Oct (2.9) with 25% Reg, 10% Polypropylene	Water	0-1
26	Oct (2.9) with 28% Reg	DMSO	5-20
26	Oct (2.9) with 28% Reg	80/20, AN/H ₂ O	2-5
26	Oct (2.9) with 28% Reg	Water	0-1
Comp. 3	Corning 3095	DMSO	~5,000*
Comp. 3	Corning 3095	80/20, AN/H ₂ O	~5,000*
Comp. 4	Zymark P/N 62367	DMSO	~5,000*
Comp. 5	Costar 6569	DMSO	15
Comp. 5	Costar 6569	80/20, AN/H ₂ O	7
Comp. 6	Ultra-Plate	DMSO	~5,000*

* AN=acetonitrile.

**Indicates that sample fully dissolved in extractant and exceeded the measurable range of the instrument. Values were calculated by mass balance before and after extraction.

5

A significant value of the present invention is the ability to limit the contamination of samples through extraction of cover tape components by common solvents. This value of the present invention is shown in Table 3.

WHAT IS CLAIMED IS:

1. An analytical receptacle comprising a surface having at least one reservoir therein and a cover tape adhered to the surface; wherein the cover tape comprises a backing and an adhesive coated on at least one major surface of the backing and in
5 contact with the receptacle surface; wherein the adhesive comprises a poly(alpha-olefin) polymer.
2. The analytical receptacle of claim 1 wherein the reservoir is in the form of a well or channel.
3. The analytical receptacle of claim 1 wherein the analytical receptacle comprises a
10 substantially continuous tape.
4. The analytical receptacle of claim 1 wherein the adhesive is pattern coated on the backing of the cover tape.
5. The analytical receptacle of claim 1 wherein the adhesive is a pressure sensitive adhesive.
- 15 6. The analytical receptacle of claim 5 wherein the adhesive is crosslinked.
7. The analytical receptacle of claim 6 wherein the adhesive is crosslinked using E-beam radiation.
8. The analytical receptacle of claim 1 wherein the poly(alpha-olefin) polymer is prepared from monomers comprising about 85 mole percent to about 100 mole
20 percent of one or more C6 to C10 alpha-olefin monomers, and about 15 mole percent to 0 mole percent of one or more polyene monomers, with the proviso that the mole percentages of all monomers sum to 100.
9. The analytical receptacle of claim 8 wherein the adhesive is prepared from a composition comprising the poly(alpha-olefin) polymer and a photoactive
25 crosslinking agent.
10. The analytical receptacle of claim 1 wherein the poly(alpha-olefin) polymer is prepared from monomers comprising about 70 mole percent to about 99 mole percent of one or more C6 to C12 alpha-olefin monomers and about 1.0 mole percent to about 30 mole percent of one or more C2 to C5 alpha-olefin monomers.

11. The analytical receptacle of claim 10 wherein the adhesive is prepared from a composition comprising the poly(alpha-olefin) polymer and a photoactive crosslinking agent.
12. The analytical receptacle of claim 1 wherein the reservoir includes a powder therein during use.
13. The analytical receptacle of claim 1 wherein the reservoir includes a liquid therein during use.
14. The analytical receptacle of claim 13 wherein the liquid comprises dimethyl sulfoxide, water, acetonitrile, methanol, ethanol, or mixtures thereof.
15. The analytical receptacle of claim 1 comprising a microtiter plate.
16. The analytical receptacle of claim 1 comprising a microfluidic device comprising a substrate and one or more channels therein.
17. The analytical receptacle of claim 1 comprising a substantially continuous polymeric strip comprising a plurality of reservoirs at predetermined intervals along its length.
18. The analytical receptacle of claim 17 wherein the reservoirs are uniformly spaced.
19. An analytical receptacle comprising a surface having at least one reservoir therein and a cover tape adhered to the surface; wherein the cover tape comprises a backing and an adhesive coated on at least one major surface of the backing and in contact with the receptacle surface; wherein the adhesive comprises a crosslinked poly(alpha-olefin) polymer; and further wherein the one or more reservoirs includes a liquid.
20. The analytical receptacle of claim 19 comprising a microliter plate.
21. The analytical receptacle of claim 19 comprising microfluidic device comprising a substrate and one or more channels therein.
22. The analytical receptacle of claim 19 comprising a substantially continuous polymeric strip comprising a plurality of reservoirs at predetermined intervals along its length.
23. The analytical receptacle of claim 19 wherein the reservoir is in the form of a well or channel.

24. An analytical receptacle comprising: a substantially continuous polymeric strip having a surface and at least one reservoir therein; and a cover tape adhered to the surface; wherein the cover tape comprises a backing and an adhesive coated on at least one major surface of the backing and in contact with the receptacle surface; and further wherein the adhesive comprises a crosslinked poly(alpha-olefin) polymer.
- 5
25. The analytical receptacle of claim 24 wherein the reservoir is in the form of a well or channel.

INTERNATIONAL SEARCH REPORT

International Application No.
PCT/US 99/12686

A. CLASSIFICATION OF SUBJECT MATTER

G01N35/00

According to International Patent Classification (IPC) or to both national classification and IPC 7

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

G01N35/00, B65D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4883642 A (BISCONTE) 28 November 1989, cited in the application.	1, 24
P, A	PATENT ABSTRACTS OF JAPAN, vol. 8, no. 056, 31 August 1999; & JP 11 139488 A (LINTEC CORP.) 25 May 1999, abstract.	1, 24



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents :

- * "A" document defining the general state of the art which is not considered to be of particular relevance
- * "E" earlier document but published on or after the international filing date
- * "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- * "O" document referring to an oral disclosure, use, exhibition or other means
- * "P" document published prior to the international filing date but later than the priority date claimed

- * "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- * "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- * "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- * "&" document member of the same patent family

Date of the actual completion of the international search
24 November 1999

Date of mailing of the international search report
20. 12 1999

Name and mailing address of the ISA
European Patent Office, P.O. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl,
Fax (+ 31-70) 340-3016

Authorized officer

NARDAI e.h.

ANHANG

ANNEX

/ INEXE

zum internationalen Recherchen-
bericht über die internationale
Patentanmeldung Nr.

to the International Search
Report to the International Patent
Application No.

au rapport de recherche inter-
national relatif à la demande de brevet
international n°

PCT/US 99/12686 SAE 239336

In diesem Anhang sind die Mitglieder
der Patentfamilien der im obenge-
nannten internationalen Recherchenbericht
angeführten Patentdokumente angegeben.
Diese Angaben dienen nur zur Unter-
richtung und erfolgen ohne Gewähr.

This Annex lists the patent family
members relating to the patent documents
cited in the above-mentioned inter-
national search report. The Office is
in no way liable for these particulars
which are given merely for the purpose
of information.

La présente annexe indique les
membres de la famille de brevets
relatifs aux documents de brevets cités
dans le rapport de recherche inter-
national visé ci-dessus. Les renseigne-
ments fournis sont donnés à titre indica-
tif et n'engagent pas la responsabilité
de l'Office.

Im Recherchenbericht angeführtes Patentdokument Patent document cited in search report Document de brevet cité dans le rapport de recherche	Datum der Veröffentlichung Publication date Date de publication	Mitglied(er) der Patentfamilie Patent family member(s) Membre(s) de la famille de brevets	Datum der Veröffentlichung Publication date Date de publication
US A 4883642	28-11-1989	CA A1 1272669 DE C0 3569483 EP A1 186676 EP B1 186676 FR A1 2565350 FR B1 2565350 JP T2 61502382 WO A1 8505563	14-08-1990 24-05-1989 09-07-1986 19-04-1989 06-12-1985 10-10-1986 23-10-1986 19-12-1985
JP A2 11139488	25-03-1999	keine - none - rien	

THIS PAGE BLANK (USPTO)